SULFATE SOILS STABILIZATION WITH MAGNESIUM-BASED BINDERS

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ABSTRACT

Sulfate soils' stabilization is a very interesting subject with technical, economic and environmental implications. The difficulty of their stabilization is due to the fact that the usual stabilizer additives are based on calcium. In these soils, sulfate combines with the calcium from the additive and the aluminum from the clay, resulting in a highly hydrated expansive mineral named ettringite. This provokes the swelling of the treated material and even its destruction. This study analyzes the result of the substitution of the calcium based additives by one alternative additive based on magnesium, an industrial byproduct named PC-8, in the stabilization of five different sulfate soils. From a mechanical point of view soils treated with PC-8 reached similar resistance values to the lime treated ones, of about 2-3 MPa for 4% dosage and 2-5 MPa for 8% dosage, being usually better with the PC-8 results than with the lime ones. When PC-8 was combined with GGBS the resistance values increased up to 11-13 MPa and the lime-GGBS reached the 6-7 MPa. The natural swelling of the soils treated with PC-8 decreased substantially and maintained constant even for immersion at long-term. In the case of the soils treated with lime, long-term swelling increased up to very high values even in the case of soils without natural swelling. XRD analysis of these samples demonstrated the existence of ettringite in 4 of the 5 soils when they were treated with lime and there was not expansive minerals in the PC-8 treated soils, agreeing with the swelling observed behavior of the soils when treated with both additives.

HIGHLIGHTS

There is a potential in using magnesium oxide as a sulfate soils stabilizer.

Magnesium oxide demonstrated a better ability as pozzolanic activator to GGBS than lime.

The long-term swelling test was a good estimator of the development of expansive minerals.

XRD results agree with the indirect estimation of the presence of expansive minerals.

All the lime and none of the PC-8 treated soils, showed expansive behavior.

KEYWORDS

Sulfate soil; soil stabilization; valorization; construction material; ettringite

INTRODUCTION

Soils' stabilization based on lime or cement is a widely used technique to improve the soils' properties as a construction material. The use of these additives is particularly suitable in clayey soils, which usually have inadequate properties like high plasticity, bad workability as well as low bearing capacity (Guney et al., 2007; Lin et al., 2007; Göktepe et al., 2008). Clayey soils consist in aluminosilicate mineral layers with negative surface charge that, by electrostatic repulsion, maintain an open structure. With the addition of lime, Ca^{2+} ions fixation takes place, electrostatic charges of the clay layers are balanced and electrochemical repulsion forces between them are reduced. This causes the adhesion of the clay particles in flocs where the clay layers are now linked by Ca^{2+} ions, resulting in a soil with improved properties: a more granular structure, higher permeability and lower plasticity (Kinuthia et al., 1999; Lin et al., 2007). In addition, the lime's hydration releases OH^- ions that increase the pH up to about 12.4. Under these conditions pozzolanic reactions take place in the soil: Aluminum (A) and silicon (S) from the clay matrix are solubilized and combine with the available calcium (C) generating hydrated (H)

cementitious compounds schematically named CSH, CAH and CSAH (Nalbantoglu, 2004; Guney et al., 2007; Yong and Ouhadi, 2007; Chen and Lin, 2009). These compounds are responsible of the improvement of the mechanical properties of the stabilized soil upon curing time, as pozzolanic reactions develop. The kinetics of these reactions depend on the amount and availability of the concerned oxides and they can last even years (Wild et al., 1998). The clay matrix usually has enough aluminum and silicon, which allows the use of lime instead of cement, being a cheaper and effective treatment (Chen and Lin, 2009). In those soils where enough silicon and aluminum are not available, they have to be added with the additive. In these cases it is recommended the use of products like Ordinary Portland Cement (OPC), rich in available SiO_2, Al_2O_3 and CaO, that provides the needed oxides, shortening also the reaction time (Wild et al., 1998; Degirmenci et al., 2007). The improvement of the obtained properties will depend on the quantity and richness of the additive, the soil's mineralogy, its particles' size and shape and the curing conditions (Misra et al., 2005; Yarbasi et al., 2007; Göktepe et al., 2008).

Despite the demonstrated technical, economic and environmental suitability of the use of calcium based additives as stabilizers for soils, this type of treatment may cause adverse effects in some soils and even lead to the total destruction of the stabilized material. One of the most common causes of this type of failure is the presence in the stabilized soil of sulfate (SO_4^{2-}). Sulfate is a very common oxide as well as in many natural soils around the world as in industrial wastes. Other times it may stem from the water used for compaction, or arise from infiltration from the surrounding terrain. Whatever its origin, the reaction of the calcium supplied by the lime or cement, aluminum, silicon and the sulfate all together in the presence of water, causes the formation of expansive minerals, the most common being a highly hydrated and expansive crystalline mineral named ettringite [$Ca_6Al_2(SO_4)_3(OH)_{12}\cdot 26H_2O$] (Crammond, 2002; Nobst and Stark, 2003;

Ciliberto et al., 2008; Norman et al., 2013). The mechanics of the formation of ettringite are not well established (Mohamed, 2000), although the conditions for its formation are known (Ouhadi and Yong, 2003), which are: (1) High pH, (2) presence of soluble Al, (3) presence of soluble Ca, (4) presence of soluble sulfate and (5) availability of water. It is also known that the rate of ettringite formation is accelerated by high temperatures (Rajasekaran, 2005). Talero (2005) and Rahhal and Talero (2014) pointed out different formation rates, shape and size of the ettringite's crystals based on different reactive aluminum compounds from OPC (C_3A) and pozzolanic additions ($Al_2O_3^{r-}$). Given the right conditions, ettringite formation is the dominant reaction, even appearing during the mixing of the materials. Mohamed (2000) determined the total time of formation of ettringite in a natural marly soil within 48 hours. In their experiments Ouhadi and Yong (2003) and (2008) established the formation of this mineral over one month in one case, and between the mixture of the soil with lime and 48 hours in another experiment. From a chemical point of view, the reactions involved in the formation of ettringite are (Ouhadi and Yong, 2008):

$$CaO + H_2O \rightarrow Ca(OH)_2$$

$$Ca(OH)_2 \rightarrow Ca^{2+} + 2(OH)^-$$

$$Al_2Si_4O_{10}(OH)_2 \cdot nH_2O + 2(OH)^- + 10H_2O \rightarrow 2\left\{2Al(OH)_4^- + 4H_4SiO_4\right\} + nH_2O$$

$$M_xSO_4 \cdot nH_2O \rightarrow XM^y + SO_4 + nH_2O$$

where

$$x = 1, y = 2$$

or

$$x = 2, y = 1$$

$$6Ca^{2+} + 2Al(OH)_4^- + 4OH^- + 3(SO_4^{2-}) + 26H_2O \rightarrow Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O$$

A second way for the ettringite formation has been observed in OPC mortars and concretes where, after long curing periods in wet conditions, sulfate attacks the CAH, generating what is commonly refered to as "delayed ettringite", which provokes the same undesirable effects in the cementitious materials (Lee et al., 2005; Escadeillas et al., 2007; Ciliberto et al., 2008; Chen and Jianj, 2009; Pavoine et al., 2012; Nguyen et al., 2013). The swelling capacity of the soil in which ettringite develops, and therefore its undesirable effects, are produced during the formation of this mineral. Once ettringite is present in the soil, it is stable and even contributes to improve bearing capacity (Kinuthia et al., 1999; Rajasekaran 2005; Min et al., 2008).

One way to reduce the formation of ettringite in a stabilized sulfate soil is the partial substitution of lime or cement by Ground Granulated Blastfurnace Slag (GGBS). GGBS contributes with a large amount of quickly available aluminum and silicon both of which react with the available calcium, expending it for the cementitious gels formation, avoiding the formation of ettringite. This protective effect against sulfate attack is especially intense for high replacement ratios of lime with GGBS (about 83%). Furthermore, GGBS also produces a denser cementitious matrix, reduces the permeability and hence the water availability, increasing the durability against the internal and external sulfates' attack (Wild et al., 1998; Tasong et al., 1999; Obuzor et al., 2012). However this way of limiting the sulfate attack must to be used carefully because all the oxides needed for the ettringite formation remain in the treated material (Celik and Nalbantoglu, 2013).

Another probable way to stabilize the sulfate soils can be the replacement of calcium based additives by other metal cations, such as magnesium. Xeidakis (1996-a) and (1996-b) demonstrated that the $Mg(OH)_2$ fixing in the expansive clay layers is more rapid than that $Ca(OH)_2$, resulting also in the clay's flocculation. He also proved the $Mg(OH)_2$

145 capacity to increase the soil's pH and the theoretical possibility to generate Mg based
146 cementitious gels.
147 The lack of guaranties about the sulfate soils stabilization with calcium additives, the
148 magnesium capacity to flocculate clays and its eventually cementing properties, both
149 make the magnesium-based additives a priority research subject.
150 This study analyzes the possibility to stabilize sulfate-containing soils with magnesium-
151 based additives instead the calcium-based ones, taking into account the capacity to create
152 cementitious gels as well as the reduced potential for the formation of expansive minerals
153 due to the stabilization of both additives.
154

155 **2. MATERIALS AND METHODS**

156 2.1. MATERIALS

157 Three natural soils (I, II and III) and two based in waste artificial soils (IV and V) were
158 considered in this study. Table 1 shows the characterization of the analyzed soils from a
159 soil mechanics and chemical points of view. All the laboratory test were carried out in
160 accordance with Spanish and European Standards, mineralogical compositions were
161 estimated by X-Ray Diffraction (XRD) analysis based on the chart proposed by Al-
162 Rawas (1999) and the soils composition, expressed as their most significant oxides, were
163 based on X-Ray Fluorescence (XRF) analysis.
164

165 TABLE 1
166

167 Soils I, II and III were obtained from different geological formations across the Ebro's
168 Valley in the North of Spain. They are expansive clayey soils from different geological
169 terciary structures with different sulfate contents.

Soil IV comes from the fraction smaller than 5 mm, obtained from crushing of old concrete structures in a building site in the city of Pamplona (Spain). This recycled material contains a 5% of sulfate, mainly because of the plasterboards remains crushed together with the concrete. Soil V consists of a mix of a 70% slag from a biomass plant that burns cereal straw, with a 30% of a local marly soil to get adequate workability properties for the soils mechanical tests.

The second kind of materials considered are the following additives: (1) PC-8. This material is a by-product rich in MgO calcined during magnesite production by means of the calcination of natural $MgCO_3$ rocks up to 1,100º C. This process is carried out in a rotatory kiln with crosscurrent air circulation, which pulls dust particles along the whole kiln. So that, this dust contains $MgCO_3$ (inert), calcined MgO (reactive) and vitrified MgO (inert) particles and it is recovered by means of air cyclones. The sample used in this study was supplied by Magnesitas de Navarra S.A. Company. (2) Commercial hydrated lime CL-90-S. Rich in $Ca(OH)_2$, it has been used like reference calcium based additive for the soils treatment, and (3) GGBS. This by-product of the steel production has been used combined with PC-8 and lime as activators. The sample used was supplied by Hanson Cement Company.

Table 2 shows the additives' properties and their XRF obtained oxide composition.

TABLE 2

In the case of the soils the only pre-treatment made consisted on grinding and sieving to get particles sizes smaller than 2 mm to allow a better contact soil-additive and thus, a more efficient treatment. In the case of the additives no pre-treatment was necessary.

2.2. SAMPLES' PREPARATION

Two kind of samples were prepared during the experimental investigation: The first category are the samples to establish the mechanical properties of the tested combinations. The mixing of soils and additives was carried out in an industrial mixer until their complete homogenization. After that, water corresponding to the Normal Proctor (NP) test was added slowly to get uniform distribution. After a wet mixing time the mixes were treated in a high speed homogenizer to guarantee good of mixing and wet distribution. Once the quality of the mixes was visually verified, 65 mm diameter and 75 mm height cylindrical samples were prepared, pressing the material in a mold at 8 MPa. The samples were immediately demolded after fabrication and maintained, since the fabrication till the testing age, in a wet chamber at 20º C and 100% HR. The additives' doses were established based on the bibliography as follows (Oti et al., 2008; Seco et al., 2011a; Seco et al., 2011b; Miqueleiz et al., 2012): Soils I, II and III were treated with PC-8 at 4 and 8%, lime at 4 and 8%, PC-8 at 2% plus GGBS at 8% and lime at 2% plus GGBS at 8%. In the case of the soils IV and V, PC-8 and lime alone were not tested because of the lack of clay minerals in the crushed concrete and in the biomass slags. In these soils only PC-8 at 2% plus GGBS at 8% and lime at 2% plus GGBS at 8% were used.

The combinations with 8% of calcium and magnesium alone where picked out to state the long-term stability of the treated soils against the sulfate attack. In this case, 50 mm diameter and 2mm height samples were prepared at NP maximum density and optimum wet content inside oedometer sample rings. In these cases, as the materials quantities were small (about 200 g in each case), the mixing as well as the compaction procedures were performed manually in accordance with the Spanish standard UNE 103601.

2.3. TESTS

220 The characterization of the mechanical properties of the tested samples was carried out at
221 the ages of 1, 7, 14, 28, 56 and 90 days, according to the unconfined compressive test
222 defined in the Spanish standard UNE 103400. The long-term materials stability was
223 analyzed in two ways: On the one hand the dimensional stability of the samples in a long-
224 term swelling was determined by immersion in the oedometer test according to the
225 Spanish standard UNE 103601. On the other hand, all the samples, after the swelling test
226 were analyzed by XRD to identify any possible mineralogical changes in the samples'
227 composition, especially the development of expansive minerals.
228
229 **3. RESULTS AND DISCUSSION**
230 3.1. MECHANICAL PROPERTIES INVESTIGATION
231 Figure 1 shows the samples' mechanical properties for the five soils considered and the
232 different additives combinations.
233
234 FIGURE 1
235
236 In the soils I-III the use of 4% of lime and of PC-8 gave the lowest resistance values with
237 no significant differences among both additives. The dosage of 8% of PC-8 resulted in
238 low or moderate mechanical strength increases that always maintain under 5 MPa. In the
239 case of the lime only in soil III a small increase of resistance was observed for the 90 days
240 curing time. For the combinations with 2% of lime or PC-8 combined with 8% GGBS,
241 the resistance increases were the most significant, especially in the case of the PC-8 that,
242 in all the soils was beyond 11 MPa, with a maximum value of 13.4 MPa in soil IV. Lime
243 plus GGBS treated soils demonstrated good increase in mechanical properties but always
244 lower than for the PC-8 plus GGBS combinations, with typical values about 6-7 MPa and

245 a maximum value of 8.8 MPa in soil III at 56 days. Another meaningful data is that, in all
246 the cases, close to maximum resistance values were reached before the 28 days,
247 shortening significantly the expected curing time for soils stabilization. Finally, the lack
248 of visual damage in the specimens during the 90 days of curing should be noted. Only in
249 samples from the combinations of soil I, with 4 and 8% of lime, where some surface
250 spalling was observed (see Figure 2), but their XRD analyzes did not show any presence
251 of ettringite.
252
253 FIGURE 2
254
255 After the experimental investigations the remaining samples of all the combinations were
256 maintained in the wet chamber for observing the possible delayed appearance of damages
257 caused by expansive minerals. In soils I-VI no defects were identified but in the case of
258 soil V at 90 days small surface cracks, which did not affect the mechanical strength of the
259 samples, were observed. These cracks in the samples treated with lime continued growing
260 up till 140 days of curing, when all the samples treated with lime and with PC-8 had the
261 appearance shown in Figure 3. Despite the evidence of the existence of expansive
262 behaviour, the XRD analyses of these samples did not identify any changes in
263 mineralogical composition that supported the appearance of expansive minerals. Figure
264 4 shows the XRD difractograms of soil V and the same soil treated with 8% lime, tested
265 after the unconfined compressive test.
266
267 FIGURE 3
268 FIGURE 4
269

3.2. ANALYSIS FOR LONG-TERM DIMENSIONAL STABILITY

Following the stated methodology, three samples of each soil with 8% of lime, 8% of PC-8 and samples without treatment (control), were prepared to analyze their free swelling upon potential in immersion. Figure 5 shows the temporal evolution of the different combinations and Table 3 summarizes them.

FIGURE 5

TABLE 3

The five soils showed similar behavior. The untreated samples maintained steady swelling values except soil V. This behavior was attributed to the presence of unburned vegetal remains in the biomass slag that swelled because of their water absorption or because of the presence of calcium and sulfate from the burnt remains that could combine with aluminum oxides from the marl, to produce small amounts of ettringite. The swelling for the soils treated with PC-8, decreased significantly, even reaching acceptable values for use as construction materials. During the entire testing time, these values remained steady except for soil IV where a slight swelling was observed, probably because of the formation of small quantities of ettringite from the cement compounds and the available sulfate. The low swelling values obtained in the samples treated with PC-8 demonstrated the good properties of MgO like expansive clays floculator as well as the elimination of swelling effects, even in the case of high dosages (8%), in immersion and at long-term. In the case of the natural soils (I-III), the treatment with lime decreased their natural swelling during the first 24 hours but after that, they swelled up to values much above their natural ones. In soils I and II, the samples swelling increased during all the test duration but in the case of the soil III, its swelling reached its maximum value at the age

of 10 days remaining steady afterward, probably due to the whole sulfate's consumption because of its small amount (0.30%). Soils IV and V treated with lime also presented significant continuous swelling values during the test duration despite their lack of natural swelling.

3.3. MINERALOGIC ANALYSIS OF THE SAMPLES

After observing macroscopic swelling behavior of the treated samples, they were analyzed by XRD to establish the relationship between the observed swelling and the presence of expansive minerals. XRD demonstrated the presence of ettringite in samples of soils I, II, IV and V treated with lime. Figure 5 shows how peaks of ettringite appear in the difractograms of these soils, revealing the presence of this mineral. These results agree in all the cases with the swelling observed behavior in the dimensional stability analysis of the samples, except in the case of the soil III. This fact, together with to the small size of the ettringite's peaks in the difractograms of the other soils where this mineral was observed, suggests that the observed type of ettringite has a low crystalline structure or more probably, it appears in small amounts, possibly in specific sites of the samples, making its identification more difficult from a mineralogical point of view, even when its macroscopical effects are evident.

FIGURE 6

As it can be seen in the Figure 6, in the case of the samples treated with PC-8 no expansive mineral was observed in any difractograms. The only difference with the soils' previous XRD characterization difractograms were the magnesite ($MgCO_3$) peaks related to the

319 additive's carbonation, agreeing with the macroscopical stability observed in the samples
320 in all the cases.
321
322 **4. CONCLUSIONS**
323 The stabilization of sulfate soils is a very interesting subject because of its technical,
324 economic and environmental implications. From a technical point of view their
325 stabilization continues being a challenge because of the concurrency on them many times
326 of circumstances that make them bad construction materials and the practical
327 impossibility of their stabilization with conventional additives. In this sense, the results
328 obtained in this study have allowed to answer some of the key questions about the effect
329 of the magnesium in different kinds of sulfate soils, among them:
330 1. The treatment of the natural soils with 4 and 8% of lime or PC-8 improved their
331 mechanical properties up to usual values obtained in other clayey soils. In all the
332 cases the developed resistances was higher for the 8% dosages than for the 4%
333 ones, and for the PC-8 than for the lime, demonstrating the ability of the
334 magnesium as a clay stabilizer additive from a mechanical point of view, better
335 than the lime.
336 2. The use of a combined binder lime-GGBS or PC-8-GGBS improved in all the
337 cases the mechanical properties of the treated soils, even before the 28 days curing
338 time. PC-8-GGBS combinations reached, in all the treated soils, the best
339 mechanical results at all the curing times, demonstrating a better ability of the
340 magnesium as pozzolanic additives activator than the calcium.
341 3. None of the samples prepared for the unconfined compressive test showed
342 significant damage or lack of resistance during the 90 days of curing time in the
343 wet chamber. Only after that age did all the remaining samples of the soil V treated

with lime start to swell during the 160 days of curing time, when they were completely disintegrated. The XRD analysis of these samples didn't show the presence of any expansive minerals in them.

4. The long-term swelling test demonstrated its ability to be an indirect estimator of the potential development of expansive minerals in the treated sulfate soils. In the samples treated with 8% lime, only in the case of the soil III, the observed 7% swelling didn't agree with the ettringite detection by XRD. In the samples treated with 8% PC-8 a significant reduction of the natural swelling of the soils was observed as well as the dimensional stability of all the treated samples across the time, except in the case of the soil IV where a small swelling was observed. In nothing of the PC-8 treated samples, expansive minerals were identified by XRD analysis.

As final conclusion it can be stated that the use of magnesium-based additives can be a potentially good method for the valorization of sulfate containing materials in construction applications. Although more studies must to be carried out in this sense, the results obtained in this study allow suggest possible replacement of calcium based additives by the magnesium ones.

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462
463 **FIGURES**

464 Figure 1. Unconfined compression test results of soils.

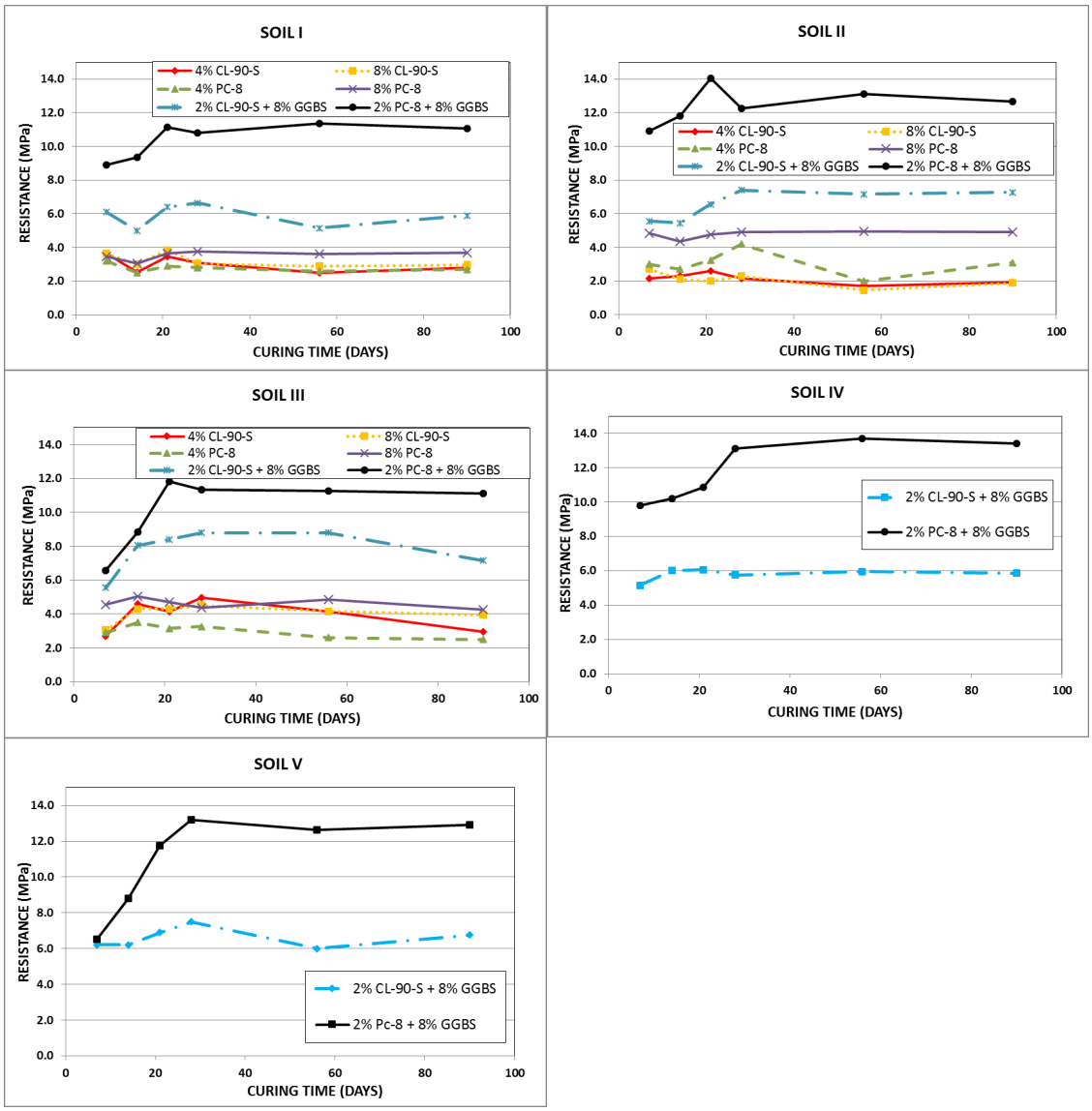


Figure 2. Soil 1 treated with 8% lime observed surface cracks at 90 days curing age.

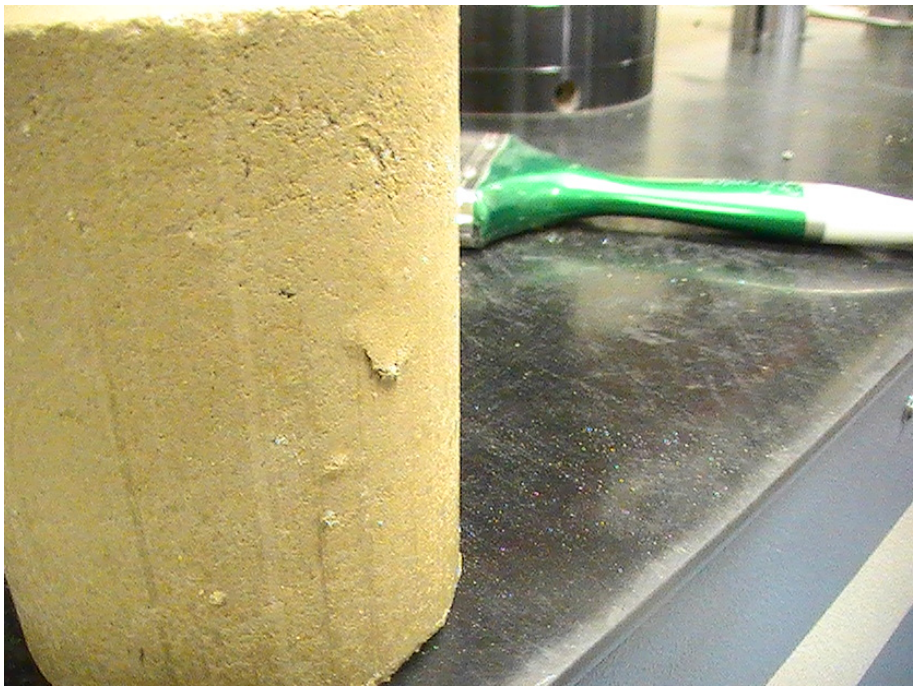


Figure 3. 140 days soil V samples. a) treated with 8% lime and b) treated with 8% PC-8.

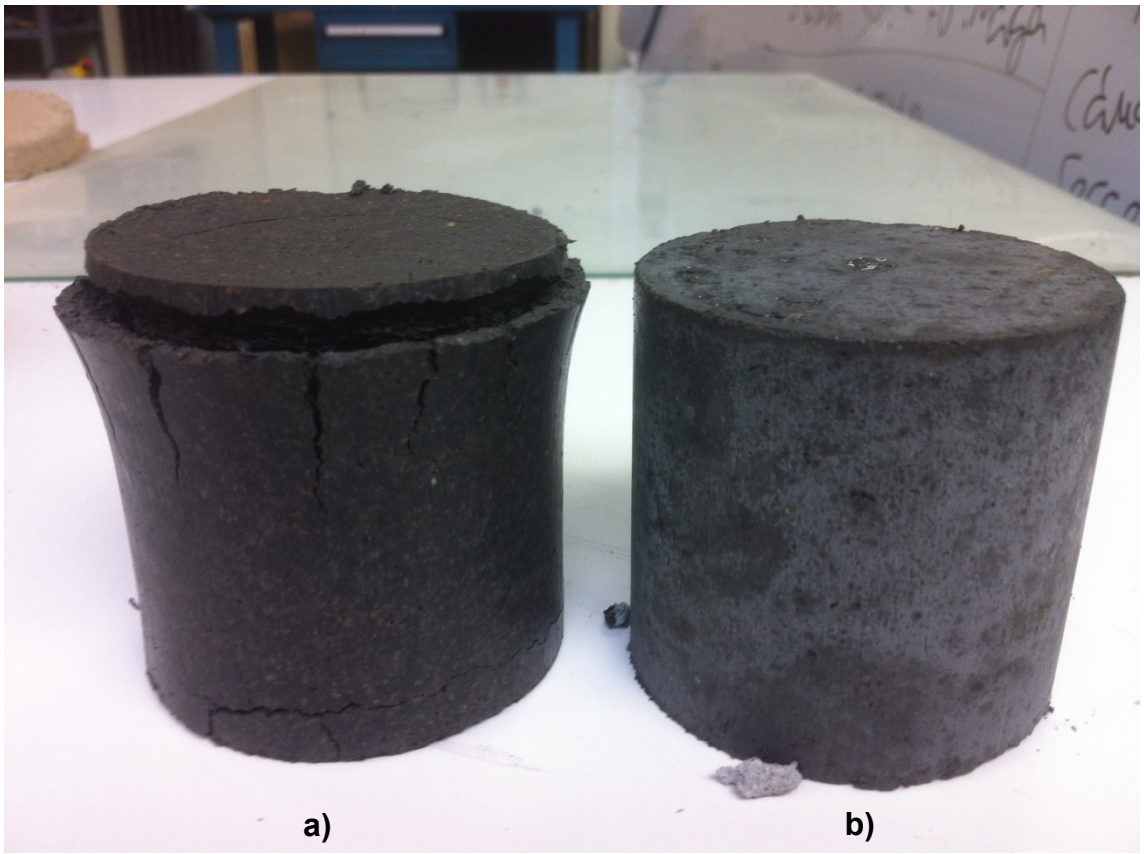


Figure 4. XRD difractograms of soil V and soil V treated with 8% lime, after the unconfined compressive test.

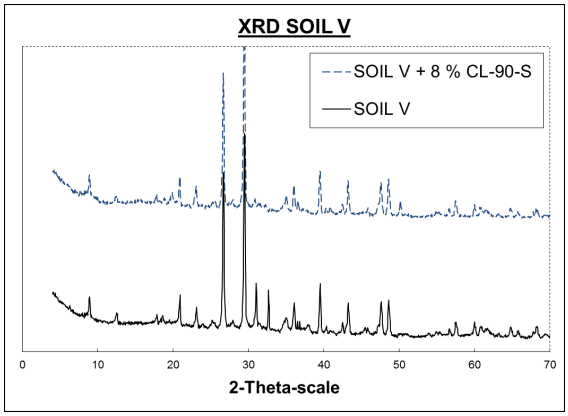
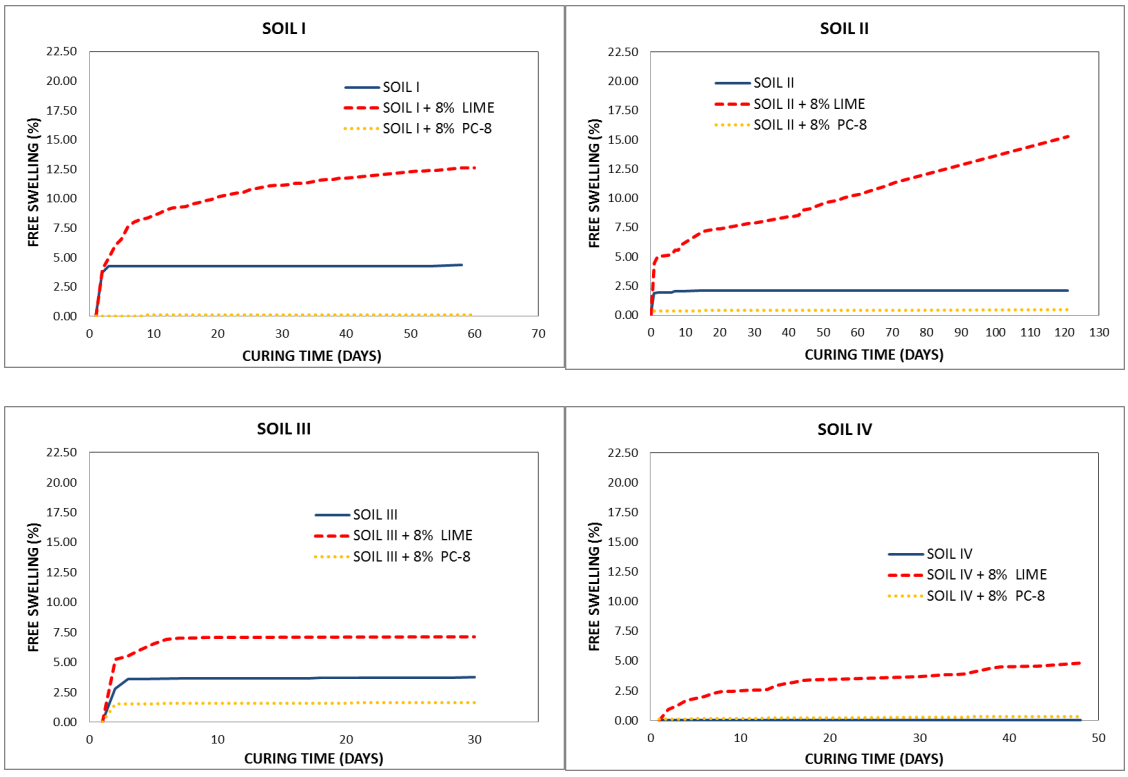
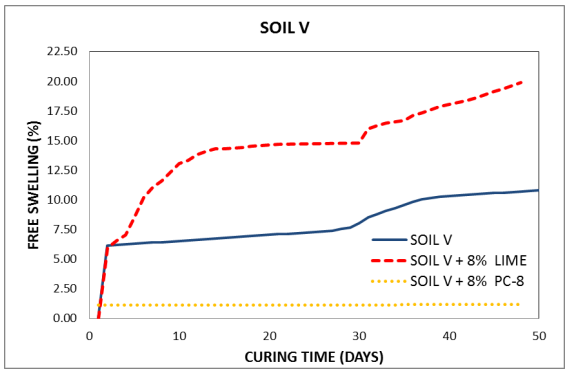


Figure 5. Long term dimensional stability analysis test results.





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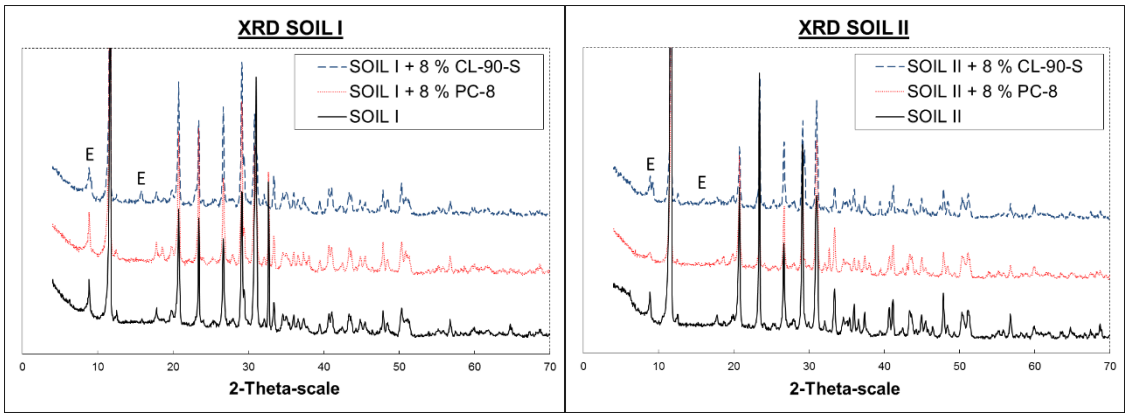
484 Figure 6. XRD of lime and PC-8 treated soils.



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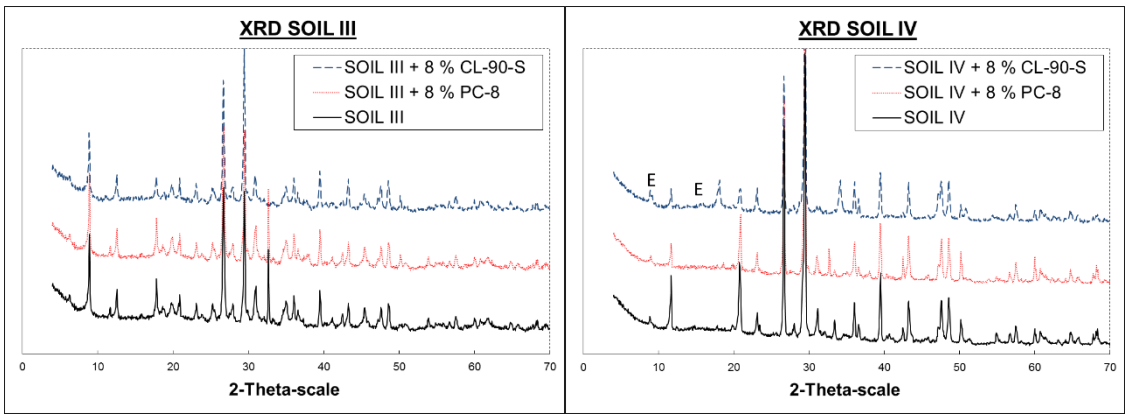

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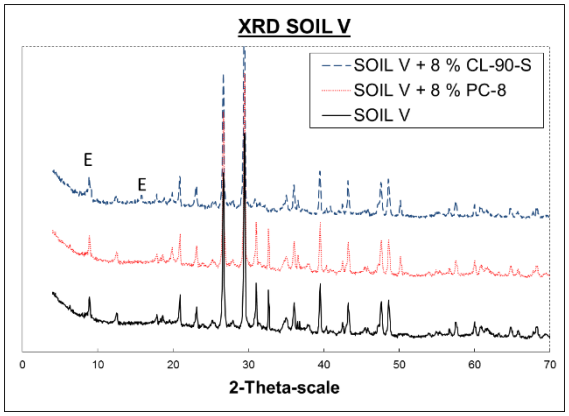

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TABLES